Designation: ~~D6514 – 03 (Reapproved 2008)~~ D6514 – 03 (Reapproved 2014)

Standard Test Method for High Temperature Universal Oxidation Test for Turbine Oils[1]

This standard is issued under the fixed designation D6514; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers a procedure for evaluating the oxidation of inhibited lubricants in the presence of air, copper, and iron metals.

1.2 This test method was developed and is used to evaluate the high temperature oxidation stability and deposit forming tendency of oils for steam and gas turbines. It has been used for testing other lubricants made with mineral oil and synthetic basestocks for compressors, hydraulic pumps, and other applications, but these have not been used in cooperative testing.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* IDENTIFIED HAZARDOUS CHEMICALS ARE LISTED IN SECTION 7. BEFORE USING THIS TEST METHOD, REFER TO SUPPLIERS' SAFETY LABELS, MATERIAL SAFETY DATA SHEETS AND OTHER TECHNICAL LITERATURE.

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A510 Specification for General Requirements for Wire Rods and Coarse Round Wire, Carbon Steel
B1 Specification for Hard-Drawn Copper Wire
D445 Test Method for Kinematic Viscosity of Transparent and Opaque Liquids (and Calculation of Dynamic Viscosity)
D664 Test Method for Acid Number of Petroleum Products by Potentiometric Titration
D943 Test Method for Oxidation Characteristics of Inhibited Mineral Oils
D974 Test Method for Acid and Base Number by Color-Indicator Titration
D3339 Test Method for Acid Number of Petroleum Products by Semi-Micro Color Indicator Titration
D4057 Practice for Manual Sampling of Petroleum and Petroleum Products
D4871 Guide for Universal Oxidation/Thermal Stability Test Apparatus
D5846 Test Method for Universal Oxidation Test for Hydraulic and Turbine Oils Using the Universal Oxidation Test Apparatus

2.2 *British Standards:*[3]

BS 1829 Specification for Carbon Steel, alternate to Specification A510

2.3 *Institute of Petroleum Standard:*[4]

IP 2546 Practice for Sampling of Petroleum Products; Alternate to Practice D4057

2.4 *ASTM Adjunct*[5]

Oxidation Cell Varnish Standard

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *Universal Oxidation Test*—the apparatus and procedures described in Guide D4871.

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[1] This test method is under the jurisdiction of ASTM Committee D02 on Petroleum ~~Products~~ Products, Liquid Fuels, and Lubricantsand is the direct responsibility of D02.09.0C on Oxidation of Turbine Oils.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from British Standards Institute (BSI), 389 Chiswick High Rd., London W4 4AL, U.K.

[4] Available from Institute of Petroleum (IP), 61 New Cavendish St., London, WIG 7AR, U.K.

[5] Available from ASTM International Headquarters. Order Adjunct No. ADJD6514. Names of suppliers in the United Kingdom can be obtained from the Institute of Petroleum. Two master standards are held by the IP for reference.

4. Summary of Test Method

4.1 After determining the viscosity at 40°C and acid number of a sample, a test specimen is stressed at 155°C for 96 h. After cooling, the test specimen is vacuum filtered for the determination of the total insolubles formed during the test. Total insolubles are reported as low, medium, or high.

4.2 The viscosity and the acid number of the filtrate are determined and compared with the initial values to ascertain any increase in those values. Both the change in acid number and the increase in viscosity at 40°C are reported.

4.3 The glass cell in which the test specimen was stressed is rinsed with heptane and dried. Residual deposits are compared with ASTM Adjunct ADJD6514,[5] and the results are reported.

5. Significance and Use

5.1 Degradation of fluid lubricants because of oxidation or thermal breakdown can result in fluid thickening or in the formation of acids or insoluble solids and render the fluid unfit for further use as a lubricant.

5.2 This test method can be used for estimating the oxidation stability of oils. It can function as a formulation screening tool, specification requirement, quality control measurement, or as a means of estimating remaining service life. It shall be recognized, however, that correlation between results of this test method and the oxidation stability of an oil in field service can vary markedly with field service conditions and with various oils.

5.3 This test method is designed to compliment Test Method D5846 and is intended for evaluation of fluids which do not degrade significantly within a reasonable period of time at 135°C.

6. Apparatus

6.1 *Heating Block,* as shown in Fig. 1, and as further described in Guide D4871, to provide a controlled constant temperature for conducting the test.

6.1.1 Test cells are maintained at constant elevated temperature by means of a heated aluminum block which surrounds each test cell. Alternate apparatus designs for sample heating and for temperature and flow control shall be acceptable, provided they are shown to maintain temperature and gas flow within the standard's specified limits.

6.1.2 Holes in the aluminum block to accommodate the test cells shall provide 1.0 mm maximum clearance for 38-mm outside diameter glass tubes. The test cells shall fit into the block to a depth of 225 ± 5 mm.

6.2 *Temperature Control System ,* as shown at lower left in Fig. 1, and as further described in Guide D4871, to maintain the test oils in the heating block at 155 ± 0.5°C for the duration of the test.

6.3 *Gas Flow Control System,* as shown at the upper left in Fig. 1, and as further described in Guide D4871, to provide dry air at a flow rate of 3.0 ± 0.5 L/h to each test cell.

6.3.1 A gas flow controller is required for each test cell.

6.3.2 Flowmeters shall have a scale length sufficiently long to permit accurate reading and control to within 5 % of full scale.

6.3.3 The total system accuracy shall meet or exceed the following tolerances:

6.3.3.1 Inlet pressure regulator within 0.34 kPa (0.05 psig) of setpoint; total flow control system reproducibility within 7 % of full scale; repeatability of measurement within 0.5 % of full scale.

6.4 *Oxidation Cell,* borosilicate glass, as shown in Fig. 2, and as further described in Guide D4871. This consists of a test cell of borosilicate glass, standard wall, 38 mm outside diameter, 300 ± 5-mm length, with open end fitted with a 34/45 standard-taper, ground-glass outer joint.

6.5 *Gas Inlet Tube,* as shown in Fig. 2, and as further described in Guide D4871. This consists of an 8-mm outside diameter glass tube, 455 ± 5 mm long, lower end with fused capillary 1.5-mm inside diameter. The capillary bore shall be 15 ± 1 mm long. The lower tip is cut at a 45° angle.

6.6 *Basic Head,* as shown in Fig. 3, and as further described in Guide D4871. This is an air condenser, with 34/45 standard-taper, ground-glass inner joint, opening for gas inlet tube, septum port for sample withdrawal, and exit tube to conduct off-gases and entrained vapors. Overall length shall be 125 ± 5 mm.

6.7 Test precision was developed using the universal oxidation/thermal stability test apparatus described in Guide D4871.[6,7] Alternate apparatus designs for sample heating, and for temperature and flow control shall be acceptable provided they are shown to maintain temperature and gas flow within the specified limits.

6.8 *Drying Oven,* explosion proof model recommended.

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[6] The sole source of supply of the standard commercial apparatus, including heating block, temperature control system, flow control system, and glassware, known to the committee at this time is Falex Corp., 1020 Airpark Drive, Sugar Grove, IL 60554-9585. Glassware for the Universal Oxidation test apparatus is also available from W.A. Sales, Ltd., 419 Harvester Ct., Wheeling, IL 60090.

[7] If you are aware of alternative suppliers, please provide this information to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee [1], which you may attend.

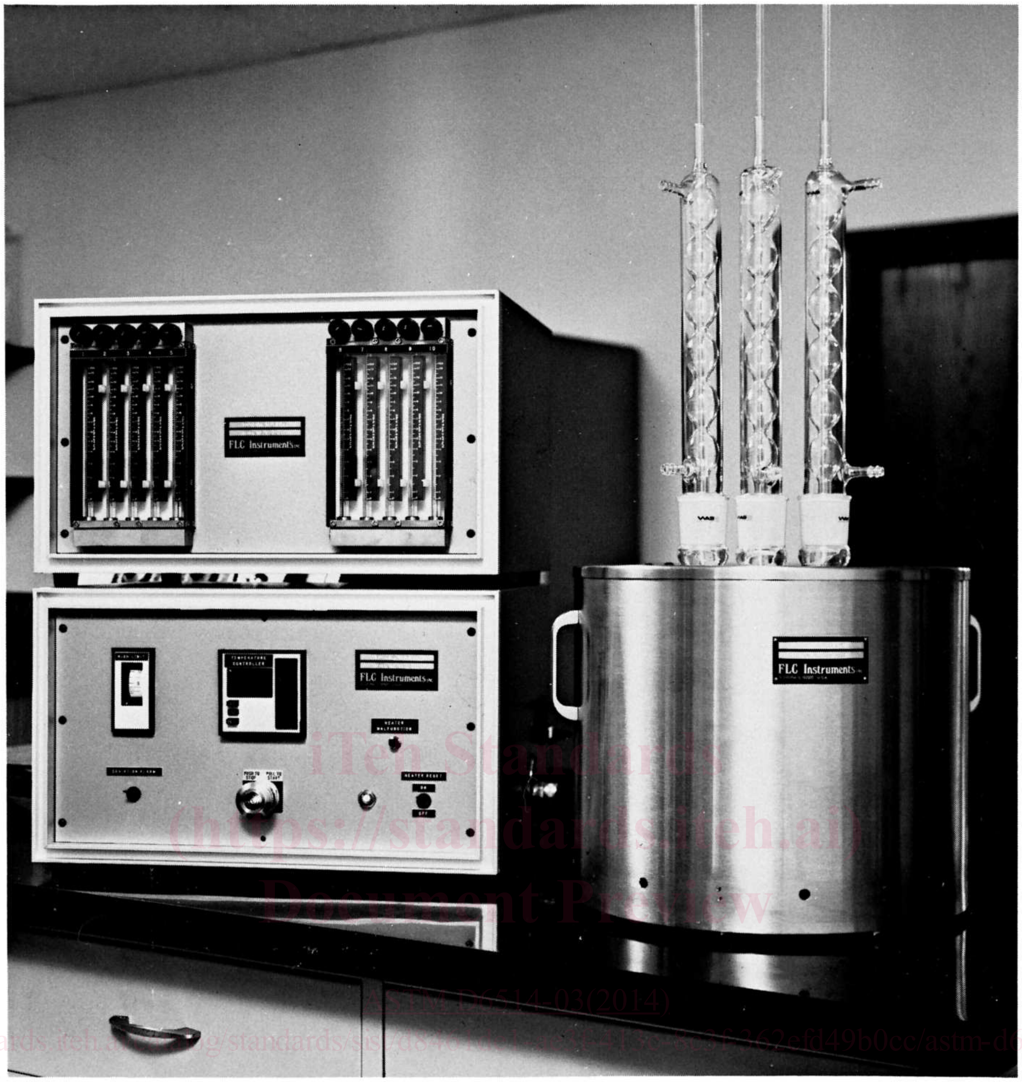

FIG. 1 Heating Block

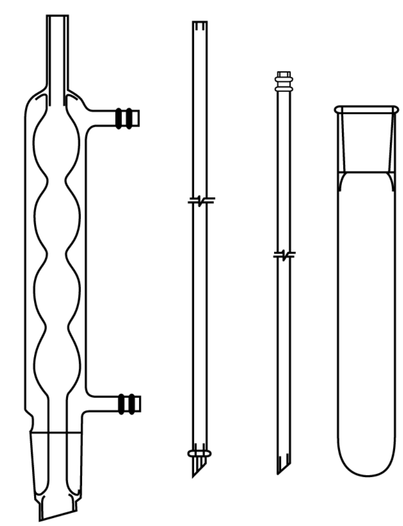

FIG. 2 Oxidation Cell

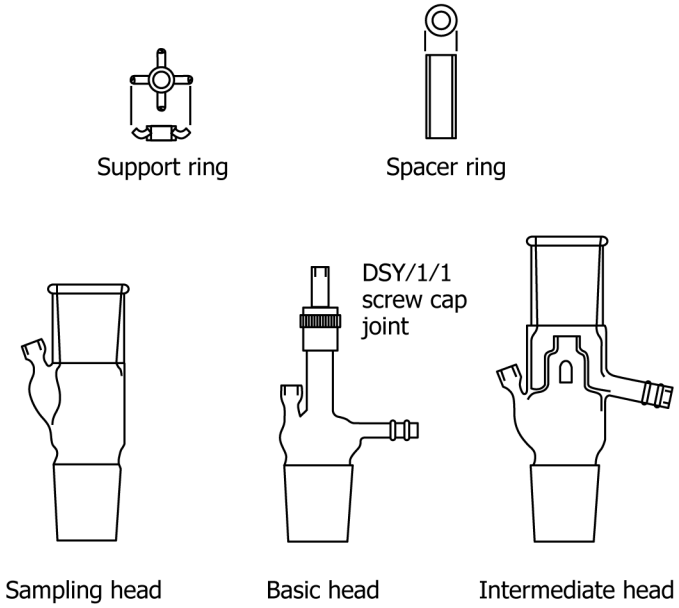

FIG. 3 Basic Head

7. Reagents and Materials

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 *Catalyst Coil (comprised of the following):*[7,9]

7.2.1 *Low-Metalloid Steel Wire,* 1.59 mm in diameter (No. 16 Washburn and Moen Gage). Carbon steel wire, soft bright annealed and free from rust, of grade 1008 as described in Specification A510, is preferred. Similar wire conforming to British Standard BS 1829 is also satisfactory.

7.2.2 *Electrolytic Copper Wire,* 1.63 mm in diameter (No. 14 American Wire Gage or No. 16 Imperial Standard Wire Gage), 99.9 % purity, conforming to Specification B1, is preferred.

7.3 *Acetone,* reagent grade. (**Warning**—Flammable. Health hazard.)

7.4 *Heptane,* reagent grade. (**Warning**—Flammable. Health hazard.)

7.5 *Propan-2-ol (iso-Propyl Alcohol) ,* reagent grade. (**Warning**—Flammable. Health hazard.)

7.6 *Isooctane,* reagent grade. (**Warning**—Flammable. Health hazard.)

7.7 *Abrasive Cloth,* silicon carbide, 100-grit with cloth backing.

7.8 *Whatman Filter Paper,* No. 41, 47–mm diameter.

7.9 *Membrane Filters,* white, plain, 47 mm in diameter pore size 8 µm. Millipore SC membrane filters (MF-type, cellulose ester),[7,10] or equivalent have been found satisfactory.

7.10 *Air,* dry.

7.11 *Cleaning Reagent,* either Nochromix[7,11] (**Warning** —Health hazard. Corrosive. Harmful if inhaled) or Micro[7,12] solution.

NOTE 1—Because of extreme hazards, chromic acid cleaning solution is not recommended.

[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For Suggestions on the testing of reagents not listed by the American Chemical Society, see *Annual Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

[9] The sole source of supply of prepared catalyst coils for use with this test method known to the committee at this time is C & P Catalyst, P.O. Box 520984, Tulsa, OK 74152.

[10] The sole source of supply of the apparatus known to the committee at this time is Millipore Filter Corp., Bedford, MA.

[11] The sole source of supply of Nochromix Reagent known to the committee at this time is Godax Laboratories, 720-B Erie Ave., Takoma Park, MD 20192.

[12] The sole source of supply of Micro known to the committee at this time is International Products Corp., P.O. Box 70, Burlington, NJ 08016.